

# PATENT SPECIFICATION

(11) 1 405 983

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- (21) Application No. 10179/73 (22) Filed 2 March 1973  
 (31) Convention Application No. 22143/72 (32) Filed 3 March 1972 (19)  
 (31) Convention Application No. 24164/72 (32) Filed 9 March 1972  
 (31) Convention Application No. 40187/72 (32) Filed 21 April 1972 in  
 (33) Japan (JA)  
 (44) Complete Specification published 10 Sept. 1975  
 (51) INT CL<sup>2</sup> C08K 5/52; C08L 67/02 // C07F 9/12; C08G 79/04;  
 (C08L 67/02, 85/02)  
 (52) Index at acceptance  
 C3R 3C 3N1 9NP 9PX C10 C12 C13P C23 C33B C8R  
 L1B L2C1 L4C L5D L6D  
 C2P 1L1 3B12B 3B15A 3B18C 3B20 3B21 7



## (54) FIREPROOF POLYESTER COMPOSITION

(71) We, MITSUBISHI RAYON CO. LTD, of 8, Kyobashi-2-chome, Chuo-ku, Tokyo, Japan, a corporation organized under the laws of Japan, do hereby declare the invention for which we pray that a Patent may be granted to us, and the method by which it is to be performed to be particularly described in and by the following statement:—

This invention relates to a fire-resistant polyester composition and more particularly it concerns a fire-resistant polyester composition which shows extremely small reduction of polymerization degree during melt-molding and which is excellent in weather resistance.

Polyesters mainly composed of polyethylene terephthalate have useful properties for being molded into fibers or films and have been widely used in industry. However, they are highly inflammable and have limitations in their use from the point of safety.

Therefore, if polyesters can be rendered fireproof without damaging the excellent physical and chemical properties which they normally possess, not only can they be widely used for clothes, interior furnishing materials, and electrical or building materials, but also they are preferred from a safety point of view.

As a method for fireproofing polyesters, there has been a method according to which the fireproofing is imparted to them in the form of final products such as woven or knitted fabric, namely, by the so-called after-processing. However, this method has the various defects that the treatment is generally complicated; the products are inferior in fastness to washing; hand of the products is markedly damaged; and fireproofing agents often have unfortunate side effect on the human body when they are used.

Therefore, it is desired to give fireproof properties to polyesters by adding fireproofing agents to polymer and at the polycondensation stage or the stage of melting and blending of the polymers to improve the above defects.

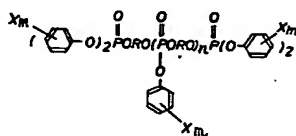
In general, it is well known that halogen-containing compounds and phosphorus-containing compounds are effective as fireproofing agents for polyesters. Some examples of halogen compounds are tetrabromo (or tetrachloro)phthalic anhydride or cyclic condensates of said anhydride and ethylene glycol, tetrabromobisphenol A or condensates thereof with a dibasic acid, hexabromobenzene and hexabromobiphenyl, and some examples of phosphorus-containing compounds are haloalkyl or haloaryl esters of phosphoric acid such as tribromopropyl phosphate, triphenyl phosphate, tris-(bromo or tribromophenyl)phosphate, tris-(chloro or trichlorophenyl)phosphate, and cyclic phosphonates.

These conventionally known fireproofing agents can give satisfactory fireproofing to polyesters, but they have various defects in that they cause coloration and decomposition of polyesters when the polyesters are melt-molded, e.g. melt-spun and cause reduction in polymerization degree and melt viscosity of polymers, whereby spinning becomes difficult or impossible or fastness to weather of the formed filaments is extremely lowered.

The inventors have accomplished this invention in view of the fact that among the known fireproofing agents, aryl esters of phosphoric acid have much excellent fireproofing and heat resistant properties and relatively good weather resistance.

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That is, this invention relates to a polyester composition comprising a synthetic linear polyester and 0.2 to 3.5% by weight (in terms of phosphorus content) of the polyester of a phosphorus compound represented by the general formula:

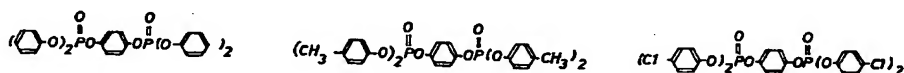


5 (wherein R being divalent is  $\text{---}\text{C}_6\text{H}_4\text{---}$ ,  $\text{---}\text{C}_6\text{H}_3\text{---}$ ,  $\text{---}\text{C}(\text{CH}_3)_2\text{---}$ ,  $\text{---}\text{CH}_2\text{---}$  and  $\text{---}\text{C}_6\text{H}_4\text{---}$ , 5

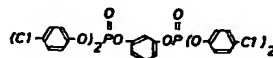
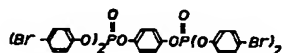
X is H, CH<sub>3</sub>, Br, or Cl, m is 1 or 2 and n is 0 or an integer of 1 to 30).

The linear polyesters used in this invention are polyethylene terephthalate, poly-  
10 tetramethylene terephthalate, poly-1,4-cyclohexane terephthalate and copolymers there-  
of with less than 20 mole % of dibasic acids such as isophthalic acid, adipic acid,  
azelaic acid or derivatives thereof, or branched copolymers thereof with diethylene  
glycol, glycerine or pentaerythritol. 10

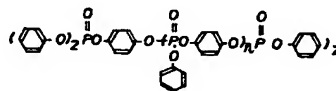
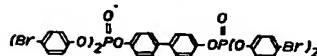
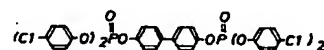
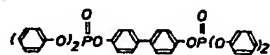
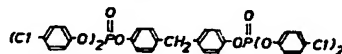
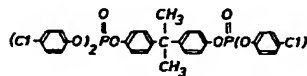
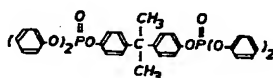
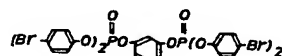
Preferred specific examples of the phosphorus compounds represented by said  
general formula and used as the fireproofing agent in this invention are as follows:



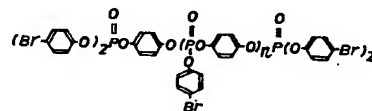
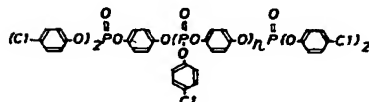
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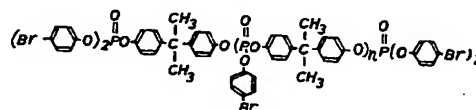
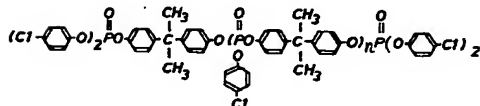
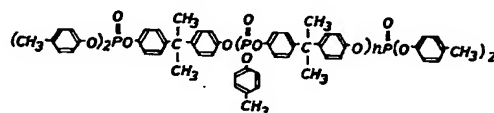
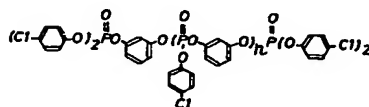
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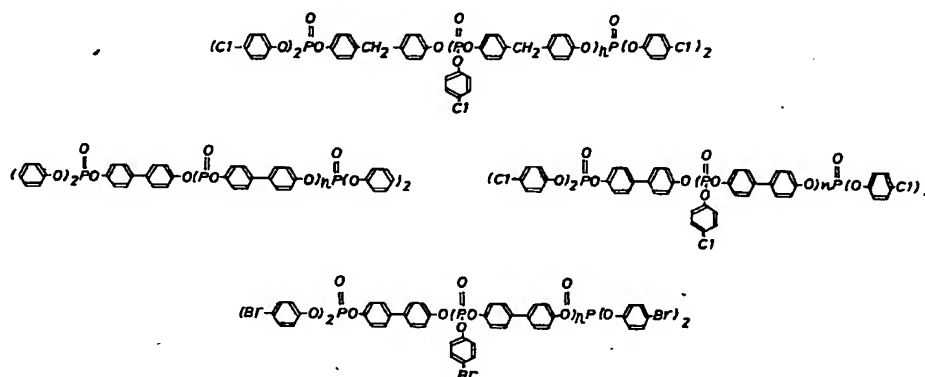


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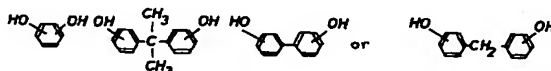
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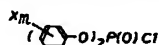


In the case of high molecular weight fireproofing agents among said compounds, when a mean molecular weight thereof exceeds 6,000, compatibility of such agents with polyesters is lowered and when the polyester is molded into fibers or film, it is apt to deteriorate. Therefore, the high molecular weight fireproofing agents have preferably a mean molecular weight of less than 6,000.

Low molecular weight fireproofing agents among said phosphorus compounds can be prepared by reacting an aromatic diol (A) represented by the general formula:



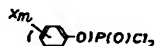
and a diarylphosphorochloridate (B) represented by the general formula



(wherein X is at least one members selected from the group consisting of H, CH<sub>3</sub>, Cl and Br and m = 1 or 2) in an organic solvent or without solvent.

Said reaction can be relatively easily carried out by heating a mixture (1:2 in molar ratio) of the diol component and the diarylphosphorochloridate in an inert gas or dried air at 150 to 250°C for several hours. Tertiary amines can be used as a catalyst for acceleration of the reaction.

Similarly, the high molecular weight fireproofing agents can be prepared in the following manner: that is, a mixture (in a given molar ratio) of the diol represented by said formula (A) and an arylphosphoryl dichloride represented by the general



(wherein X is selected from H, CH<sub>3</sub> and Br and m = 1 or 2) (C) is heated and refluxed in an organic solvent such as xylene under an inert gas or dried air for 20 to 30 hours to accomplish esterification. Thereafter, diarylphosphorochloridate (B) or phenol are added to the resultant ester and the mixture is further heated and refluxed for ten and several hours to cap both terminals of the intermediate product. Then, an aqueous solution (2 to 4 N) of NaOH is added and they are stirred to remove unreacted phenolic materials. Thereafter, the product is repeatedly and successively washed with water, dilute HCl and NaHCO<sub>3</sub> aqueous solution and then the solvent is distilled out. In this reaction, use of magnesium chloride, etc. as a catalyst is effective for increase of reaction rate.

Suitable examples of the diol component represented by the formula (A) are hydroquinone, resorcinol, 4,4'-dihydroxybiphenyl, 4,4'-methylenebisphenol, and 4,4'-methylenbis(2,6-di-tert-butylphenol).

A characteristic of the fireproofing agents in this invention is that the diol component represented by the formula (A) contains no halogen. For example, phosphorus compounds having aromatic residue such as tetrachlorohydroquinone, tetrabromobisphenol A and halogenated biphenols are extremely low in heat stability due to the

halogens substituted and cause coloration and decomposition of polyester at melt-spinning to make the spinning much difficult. On the other hand, the phosphorus compounds used in this invention hardly cause coloration even at a temperature higher than 270°C and moreover decrease of polymerization degree of the polymer is small and reduction of melt viscosity is small and sufficient spinning stability is retained.

The amount of the phosphorus compounds blended with polyesters is 0.2 to 3.5% by weight of the polyester, more preferably 0.5 to 2.2% by weight of the polyester in terms of phosphorus content. When the amount is less than 0.2% by weight of the polyester, sufficient fireproofing cannot be obtained and when more than 3.5% by weight, a significant improvement in fireproofing effect cannot be attained and the compounds cause discoloration and reduce the degree of polymerisation or reduce drastically the weather resistance of fireproofed filaments.

The time at which the fireproofing agent is to be added to the polyester is preferably after completion of the polycondensation reaction and immediately before spinning because the melting time of the fireproofing agent and polyester is preferably as short as possible. For example, the fireproofing agent can be directly added to a polymerization container after completion of the polycondensation reaction and then the mixture is spun or it can be mixed in the form of master pellets containing a large amount of the agent with a given amount of polyester in a spinning machine and then spinning can be carried out. Moreover, a method according to which the fireproofing agent is mixed in a molten state during spinning in a spinning machine is also preferable. Furthermore, in the case of composite fibers of bicomponent structure, it is possible to add the fireproofing agent to one of the components and then to carry out composite spinning.

The polyester composition of this invention may contain conventional additives, e.g. catalyst stabilizing agents, fluorescent brightening agents, and pigments.

Thus obtained polyester composition of this invention may be used for production of fireproof fibers and films in accordance with the ordinary method.

The fireproof staples or filament yarns from the composition of this invention may be woven or knitted as they are or in the form of a mixture with other fibers into fireproof curtains, carpets, children's wear, sleeping wear, bed sheets, etc. by the ordinary weaving and knitting technique.

Fibers from the composition of this invention, being different from those to which fireproofing is given by after-finishing, are given their fireproofing property uniformly at or before the stage of forming filaments. Therefore, the fibers made from the composition of this invention have various advantages that the fireproofing agent does not shed at the pre-treatment step such as dyeing step, the feel of the fibers hardly changes and they are excellent in fastness to washing.

The especially great characteristic of the polyester composition of this invention is that reduction of polymerization degree at melt molding is extremely small to such degree as that of the ordinary polyester and moreover it has remarkably good fastness to weather, so that, it has extremely high industrial value.

This invention will be illustrated in the following Examples in which "part" means "part by weight" and intrinsic viscosity  $[\eta]$  of polyester was measured in phenol-tetrachloroethane (1:1 in weight ratio) at 25°C. The evaluation of fireproofing degree was made in accordance with the following two methods.

(1) Match test method:

Fibers obtained by spinning and drawing are twisted into a string of about 3—5 mm in diameter and about 7 cm in length and the samples are vertically supported and the lower end of the samples is ignited with a match to evaluate fireproofing degree.

(2) Limited oxygen index method (LOI):

Measurement was carried out in accordance with The Oxygen Index Method of ASTM-D-2863-70. Ignition was carried out by igniting the upper part of vertically supported sample. As the samples to be measured, felt-like material of 10 cm in length, 1 cm in width and 0.3 cm in thickness and having a weight per unit area of 0.17 g/cm<sup>2</sup> which was obtained by subjecting the fibers obtained by melt spinning and drawing the composition of this invention to needle-punching was used.

Moreover, shedding of the fireproofing agent due to dry cleaning and household washing was measured in terms of fireproofing degree (LOI) after cleaning. The conditions for the cleaning were as follows:

(1) Dry cleaning:

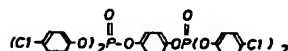
The samples were dipped in perchlene (tetrachloroethylene) at a liquor ratio of 1:20 at normal temperature for 30 minutes and then dehydrated and air-dried.

## (2) Household washing:

The samples were washed with Emul 0 (an anion surfactant, a product of Kao Sekken Co.) (2 g/l) at a liquor ratio of 1:50 at 80°C for 30 minutes by a household washing machine, then washed with water, dehydrated and dried by hot air drier at 80°C.

## Example 1.

272 Parts of bis-(p-chlorophenyl)chlorophosphate and 44 parts of hydroquinone were charged into a reactor and were allowed to react at 200°C for about 15 hours under nitrogen stream. After cooling, the reaction product was added dropwise to methanol to obtain a white precipitate, which was filtered and washed with water and recrystallized from methanol. The product had a melting point of 129 to 130°C. It was confirmed by infrared absorption spectrum and elementary analysis that said product was



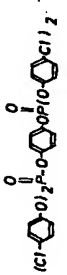
This compound and polyethylene terephthalate having an intrinsic viscosity of 0.73 were mixed in the proportion as shown in Table 1 and the mixture was vacuum dried under 1 mmHg at 130°C for 12 hours. The mixture was formed into pellets by an extruder of 40 mm in inside diameter. The resultant pellets were further vacuum dried under 1 mmHg at 130°C for 12 hours and then were spun by an extruder type spinning machine of 30 mm in inside diameter. Spinning conditions were as follows:

Diameter of nozzle .....	0.3 mm
Number of holes .....	36
(length:diameter of each hole = 2:1)	
Spinning velocity .....	1,250 m/min
Output .....	33 g/mm
Spinning temperature	
{ Barrel portion .....	275°C
{ Nozzle portion .....	280°C

Spinnability was excellent and no breakage of filaments occurred. Thus obtained undrawn filaments were drawn at a pin temperature of 90°C, a plate temperature of 160°C, a velocity of 600 m/min and a drawing ratio of 3.3 times.

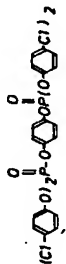
In the same manner as mentioned above, polyethylene terephthalate containing no fireproofing agent was spun. In this case, spinning temperature was 290°C at barrel portion and 290°C at nozzle portion. Various properties of thus obtained drawn filaments are shown in Table 1.

TABLE 1

<div>Fire proofing agent</div> <div>Items</div>	<div></div>		Control (no addition)
Intrinsic viscosity $[\eta]$ of pellets of raw material	0.73		0.73
Amount of fireproofing agent added (weight %)	10 (0.9% by weight in terms of phosphorus content)	15 (1.3% by weight in terms of phosphorus content)	—
Intrinsic viscosity $[\eta]$ of pellets containing fireproofing agent	0.65		0.69
Drawn filaments			
Denier	75/36	75/36	75/36
Dry tenacity	4.13	3.96	5.05
Dry extension (%)	29.8	32.4	25.9
Young's modulus	80	85	78
Birefringence	0.1362	0.1303	0.1540
$\rho$	1.3905	1.4080	1.3890

The "Dry tenacity" and "Dry extension" were measured using a "Tensilon" (Registered Trade Mark) (Model VTM-II) apparatus (manufactured by Toyo Sokki K.K.), at 20°C. 65% humidity. The length of the test filament was 2 cm and the tension speed 100%/min.

TABLE 1 (Continued)

Items	Fire proofing agent		Control (no addition)
Fireproofing of drawn filaments (LOI)		30.5	21.0
Fireproofing after 3 dry cleanings (LOI)		31.5	21.5
Fireproofing after 10 household washings (LOI)		32.0	21.0
Fastness to light (Fade-o-meter* irradiation for 80 hr.)	Coloration after irradiation	No coloration	No coloration
	DS retention rate (%) DE retention rate (%)	94 93	96 94

\* Reg. T.M. (an Ultraviolet and Carbon arc-type tester for fastness to light manufactured by Suga Tester K.K.)

#### Comparative Example 1.

5 A composition comprising 99 parts of the same polyethylene terephthalate as of  
Example 1 and 1 part of the fireproofing agent used in Example 1 (0.1% by weight in  
terms of phosphorus content) was vacuum dried under 1 mmHg at 130°C for 12 hours  
and then pelletized into pellets, which were further vacuum dried at 130°C for 12  
hours. The pellets were spun and drawn in the same manner as in Example 1. Thus  
10 obtained fibers were lightly twisted into a string of about 3—5 mm in diameter and  
about 7 cm in length. Then, the string was vertically held and the lower end thereof  
was ignited with a match to result in substantially complete combustion of the whole  
string by one ignition. The same fibers as obtained above were needle punched to pro-  
duce a felt-like sample having the size as defined before. The LOI value (A.S.T.M.)  
of this sample was 21.5.

15 Furthermore, fibers obtained from a composition (3.8% by weight in terms of  
phosphorus content) comprising 55 parts of polyethylene terephthalate and 45 parts of  
the fireproofing agent used in Example 1 in the same manner as in Example 1 were  
twisted into a string, which was vertically held. The lower end thereof was ignited with  
a match to result in utterly no combustion. The same fibers as mentioned above were  
needle punched to produce a felt-like sample of said size, which had a LOI of 39.0 and  
20 showed very high flame retardance. However, the sample subjected to a light resistance  
test by irradiation for 80 hours with Fade-o-meter was coloured yellow and showed a  
DS retention rate of 55%, and a DE retention rate of 60%, which mean that the fibers  
were markedly deteriorated.

#### Example 2.

25 The same polyethylene terephthalate as used in Example 1 and each of the follow-  
ing phosphorus compounds as fireproofing agent in the following proportions were  
charged in a glass ampoule and were vacuum dried under 1 mmHg at 130°C for 12  
hours. Thereafter, they were heated at normal pressure in nitrogen stream and at an  
outer temperature of 283°C for 60 minutes to test the heat resistance thereof. The  
results are shown in the following Table 2, from which it is recognized that use of the  
phosphorus compounds No. 2, 3 and 4 according to this invention resulted in remark-  
ably higher heat stability than the comparative phosphorus compounds No. 5, 6 and 7.



TABLE 2

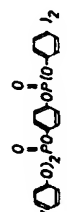
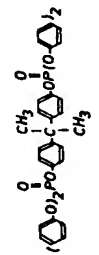
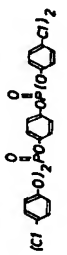
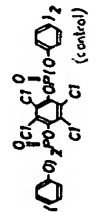
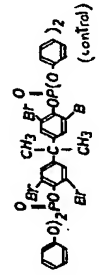
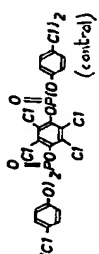
No.	Fire proofing agent	Amount of fireproofing agent blended (% by weight)	Coloring grade of polymers after subjected to heat resistant test	Intrinsic viscosity the polymer obtained
1	None	—	0.5	0.71
2		15 (1.6% by weight in terms of phosphorus content)	1.0	0.58
3		15 (1.2% by weight in terms of phosphorus content)	0.5	0.57
4		15 (1.3% by weight in terms of phosphorus content)	1.0	0.60
5	 (control)	15 (1.3% by weight in terms of phosphorus content)	4.5	0.41

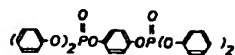
TABLE 2 (Continued)

No.	Fire proofing agent	Amount of fireproofing agent blended (% by weight)	Coloring grade of polymers after subjected to heat resistant test	Intrinsic viscosity the polymer obtained
6	 (cont'd)	15 (0.9% by weight in terms of phosphorus content)	5.0	0.35
7	 (cont'd)	15 (1.1% by weight in terms of phosphorus content)	4.5	0.40

Note: Coloring grade: 0 . . . . . colorless  
 1 . . . . . light yellow  
 2 . . . . . yellow  
 3 . . . . . light brown  
 4 . . . . . brown  
 5 . . . . . black

## Example 3.

324 Parts of diphenylchlorophosphate and 66 parts of hydroquinone were mixed to obtain

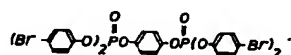


in the same manner as in Example 1.

A composition (1.6% by weight in terms of phosphorus content) comprising 85 parts of polyethylene terephthalate and 15 parts of the compound obtained as mentioned above was pelletized in the same manner as in Example 1 and then dried. Thereafter, the pellets were spun and the resultant filaments were drawn in the same manner as in Example 1. Thus obtained fibers were lightly twisted into a string of about 3—5 mm in diameter and about 7 cm in length. The string was vertically held and the lower end of the sample was ignited with a match. When the source of fire was kept away therefrom, the fire immediately went out. When said fibers as obtained above were needle punched to produce a felt-like sample having the size as defined before. LOI of the sample was 31.0. The sample subjected to a light resistance test by irradiation for 80 hours with Fade-o-meter was not substantially colored and showed a DS retention rate of 94% and a DE retention rate of 92%. That is, it has an excellent fastness to light. Furthermore, fireproofing (LOI) of the sample after subjected to 3 dry cleanings was 31.5 and LOI of the sample after 10 household washings was 32.0 and thus the sample had a good durability.

## Example 4.

258 Parts of bis-(p-bromophenyl)chlorophosphate and 33 parts of hydroquinone were mixed to obtain



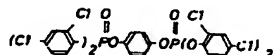
in the same manner as in Example 1. A composition (0.7% by weight in terms of phosphorus content) comprising 90 parts of polyethylene terephthalate and 10 parts of said phosphorus compound was pelletized in the same manner as in Example 1. The resultant pellets were vacuum dried under 1 mmHg at 130°C for 12 hours and then were spun. The obtained filaments were drawn.

Thus obtained fibers were twisted into a string of about 3—5 mm in diameter and about 7 cm in length. The string was vertically held and the lower end thereof was ignited with a match. Ignition was attained, but the flame immediately went out when the ignition source was kept away therefrom.

The fibers as obtained before were needle punched to produce a felt-like sample having the size as defined before. LOI of the sample was 29.0. The sample subjected to light resistance test by irradiation for 80 hours with Fade-o-meter was colored in light yellow and light resistance was relatively good. The sample showed a DS retention rate of 91% and a DE retention rate of 87% and thus had a good light resistance. LOI of the sample after subjected to 3 dry cleanings was 31.0 and that after 10 household washings was 31.5.

## Example 5.

222 parts of bis-(2,4-dichlorophenyl) chlorophosphate and 30 parts of hydroquinone were mixed to obtain



in accordance with the procedure of Example 1. A composition (1.1% by weight in terms of phosphorus) comprising 85 parts of the same polyethylene terephthalate as used in Example 1 and 15 parts of said compound was pelletized into pellets in the same manner as in Example 1 and the pellets were dried and then spun. The resultant filaments were drawn in the same manner as in Example 1.

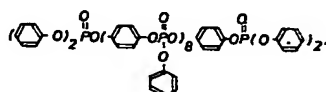
Thus obtained fibers were twisted into string of about 3—5 mm in diameter and about 7 cm in length. The string was vertically held and the lower end thereof was ignited with a match and when fire source was removed, the fire immediately went out.

The fibers as obtained before were needle punched to produce a felt-like sample having the size as defined before. LOI of the sample was 30.0 and had a high fireproof-

ing. The fibers when irradiated for 80 hours with Fade-o-meter were not substantially colored and had a DS retention rate of 88% and a DE retention rate of 89% and had a good fastness to light. Regarding washing resistance, LOI of the sample after subjected to 3 dry cleanings was 32.0 and LOI after subjected to 10 household washings was 31.5. That is, it had excellent durability.

#### Example 6.

320 Parts of phenylphosphoryldichloride, 138 parts of hydroquinone and 500 ml of xylene as a solvent were charged in a reactor and heated and refluxed for about 20 hours. Thereafter, 60 parts of phenol was added thereto and reaction was continued for further about 10 hours. After completion of the reaction, the reaction product was washed several times with a 3—4 N aqueous solution of NaOH to remove unreacted phenolic materials and the organic layer was washed several times successively with water, dilute hydrochloric acid and an aqueous solution of  $\text{NaHCO}_3$  in this order. Thereafter, the solvent was distilled out to obtain



A composition (1.2% by weight in terms of phosphorus content) comprising 90 parts of the same polyethylene terephthalate as used in Example 1 and 10 parts of said compound was pelletized in the same manner as in Example 1. The resultant pellets were dried and spun. Then the resultant filaments were drawn.

Thus obtained fibers were lightly twisted into a string of about 3—5 mm in diameter and about 7 cm in length. The string was vertically held and the lower end thereof was ignited with a match. The ignition was attained, but the fire immediately went out when fire source was removed.

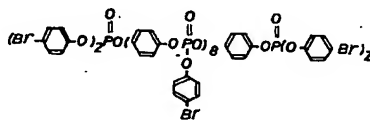
When said fibers as obtained before were needle punched to obtain a felt-like sample having the size as defined before, the felt-like sample had a LOI of 28.5, which meant good fireretardance.

Furthermore, the sample when subjected to a light resistance test by irradiation for 80 hours with Fade-o-meter was not colored at all and had a DS retention rate of 93% and a DE retention rate of 94%. Thus, it had markedly good light resistance.

Furthermore, LOI of the sample after 3 dry cleaning treatments was 29.0 and that of the sample after 10 household washings was 31.0. Thus, it had good fastness to washing.

#### Example 7.

260 Parts of p-bromophenylphosphoryl dichloride, 88 parts of hydroquinone and 500 ml of xylene as a solvent were charged in a reactor and were heated and refluxed for about 20 hours. Thereafter, 40 parts of p-bromophenol was added thereto and the reaction was continued further for about 15 hours. After completion of the reaction, the reaction product was washed several times with a 3—4 N aqueous solution of NaOH to remove unreacted phenolic materials. Then, the organic layer was separated and washed several times successively with water, dilute hydrochloric acid and an aqueous solution of  $\text{NaHCO}_3$  in that order. Thereafter, the solvent was distilled out to obtain



A composition (0.9% by weight in terms of phosphorus content) comprising 90 parts of polyethylene terephthalate and 10 parts of said phosphorus compound was pelletized into pellets. These pellets were dried, spun and drawn in the same manner as in Example 1. Thus obtained fibers were twisted into a string of about 3—5 mm in diameter and about 7 cm in length. The string was vertically held and the lower end thereof was ignited with a match. Although ignition was attained, the flame immediately went out when ignition source was removed.

Moreover, when said fibers as obtained before were needle punched to obtain a felt-like sample having the size as defined before, LOI of the sample was 31.0%, which indicated good fireretardance.

The sample when subjected to heat resistance test by irradiation for 80 hours with

Fade-o-meter was not colored at all and had a DS retention rate of 84% and a DE retention rate of 83%. Thus, it had an excellent light resistance.

The same sample after 3 dry cleaning treatments had a LOI of 30.5 and the sample after 10 house-hold washing treatments had a LOI of 32.5. Thus, it had a good washing resistance.

#### Example 8.

The same procedure as in Example 1 except that the following fireproofing agents were substituted for those used in Example 1 was repeated and the properties of the resultant fibers are shown in Table 3.

TABLE 3

No.	Fireproofing agent	Amount of the agent added (% by weight) in terms of phosphorus content	Fireproofing of drawn filaments (LOI)	Heat resistance (after irradiation for 80 hours with Fade-o-meter)	
				DS retention rate (%)	DE retention rate (%)
1		10 (1.0)	29.0	90	87
2		10 (0.9)	28.0	90	86
3		15 (1.3)	31.5	94	93
4		15 (1.0)	29.5	91	92

TABLE 3 (Continued)

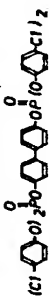

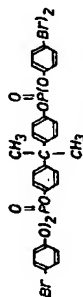
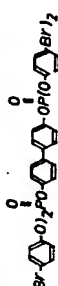
No.	Fireproofing agent	Amount of the agent added (% by weight) (% by weight in terms of phosphorus content)	Fireproofing of drawn filaments (LOI)	Heat resistance (after irradiation for 80 hours with Fade-o-meter)	
				DS retention rate (%)	DE retention rate (%)
5		15 (1.2)	31.0	90	88
6		15 (1.2)	30.5	85	87
7		15 (0.9)	29.0	83	82
8		15 (1.0)	30.0	86	84

TABLE 3 (Continued)

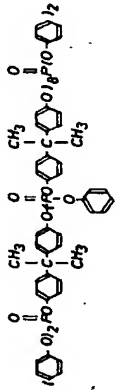
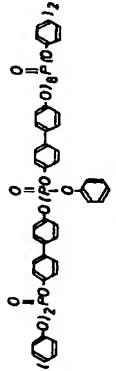
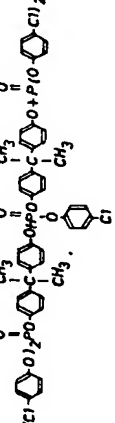
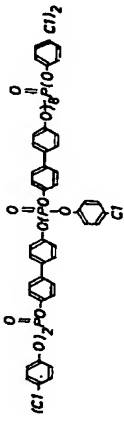
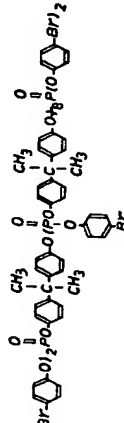
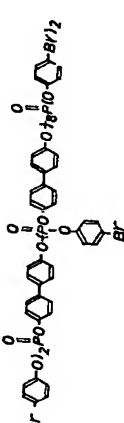
No.	Fireproofing agent	Amount of the agent added (% by weight) in terms of phosphorus content	Fireproofing of drawn filaments (LOI)	Heat resistance (after irradiation for 80 hours with Fade-o-meter)	
				DS retention rate (%)	DE retention rate (%)
9		15 (1.3)	29.5	92	93
10		15 (1.4)	30.5	90	91
11		15 (1.2)	31.5	92	85

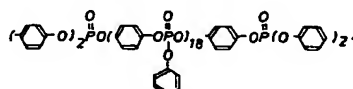
TABLE 3 (Continued)

No.	Fireproofing agent	Amount of the agent added (% by weight) in terms of phosphorus content)	Fireproofing of drawn filaments (LOI)	Heat resistance (after irradiation for 80 hours with Fade-o-meter	
				DS retention rate (%)	DE retention rate (%)
12		15 (1.3)	31.0	90	89
13		15 (1.1)	31.5	87	88
14		15 (1.0)	32.5	88	85



## Example 9.

422 Parts of phenylphosphoryldichloride, 209 parts of hydroquinone and 560 ml of tetrachloroethane as a solvent were charged in a reactor and heated and refluxed for about 27 hours. Then, 65 parts of phenol was added thereto and the mixture was further reacted for about 18 hours. After completion of reaction, the same procedure as in Example 5 was carried out and then the solvent was distilled out to obtain



A composition (1.23% by weight in terms of phosphorus content) comprising 90 parts of polyethylene terephthalate as used in Example 1 and 10 parts of said compound obtained above was pelletized in the same manner as in Example 1.

Thus obtained pellets were dried by the ordinary method and then were spun and drawn.

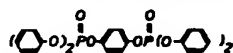
Thus obtained fibers were lightly twisted into a string of about 3—5 mm in diameter and about 7 cm in length. This was vertically held and the lower end thereof was ignited with a match. Although the ignition was attained, the flame immediately went out when the source was removed.

The fibers as obtained before were needle punched to obtain a felt-like sample having the same size as that in Example 2. LOI of thus obtained sample was measured to obtain 29.5, which meant excellent fireretardance. Moreover, this sample was subjected to light resistance test by irradiation for 80 hours with a Fade-o-meter to find substantially no coloration of the sample and the sample had a DS retention rate of 94% and DE retention rate of 92%. Thus, the sample had very good light resistance.

LOI of the same sample after subjected to 3 dry cleanings was 29.5 and that of the sample after subjected to 10 household washings was 30.5. Thus, the sample had good washing resistance.

## Example 10.

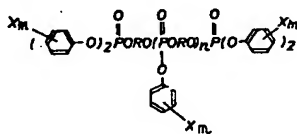
A composition (3.24% by weight in terms of phosphorus content) comprising 70 parts of polytetramethylene terephthalate and 30 parts of



was dried under 1 mmHg at 130°C for 12 hours with a revolving vacuum drier. The mixture was extruded with an extruder of 40 mm in inside diameter at a barrel temperature of 250°C and head temperature of 245°C to produce pellets, from which a plastic-like sample of 0.3 cm in thickness, 10 cm in length and 1 cm in width was prepared. Thus obtained sample was subjected to fireproof test to obtain a LOI of 40.5. The same sample was subjected to light resistance test by irradiation for 80 hours with a Fade-o-meter to cause substantially no coloration. Thus, the sample had an excellent light resistance. LOI of the polytetramethylene terephthalate itself to which no fireproofing agent was added was 27.5 (this LOI was measured in the same manner as employed above.).

## WHAT WE CLAIM IS:—

1. A polyester composition comprising a synthetic linear polyester and 0.2 to 3.5% by weight (in terms of phosphorus content) of said polyester of a phosphorus compound represented by the general formula:

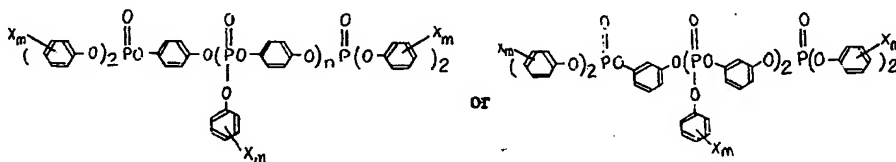


(wherein R being divalent is or ,

X is H, CH<sub>3</sub>, Br or Cl, m is 1 or 2 and n is 0 or an integer of 1 to 30).



9. A polyester composition according to Claim 1, wherein the phosphorus compound is represented by the following general formula:

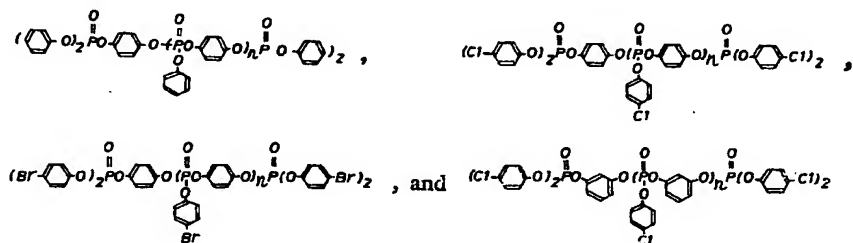


(wherein X, m and n are as previously defined).

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10. A polyester composition according to Claim 9, wherein the phosphorus compound is selected from the following group:

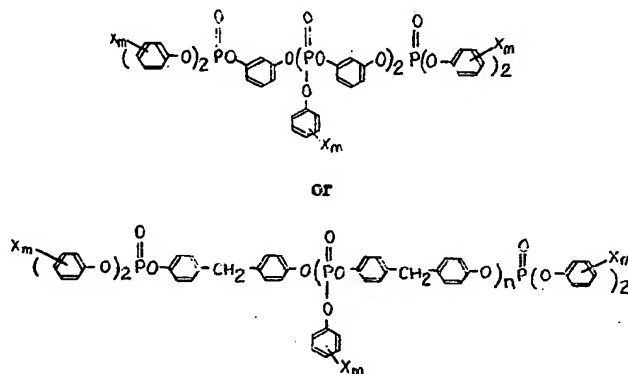
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11. A polyester composition according to Claim 1, wherein the phosphorus compound is represented by the following general formula:

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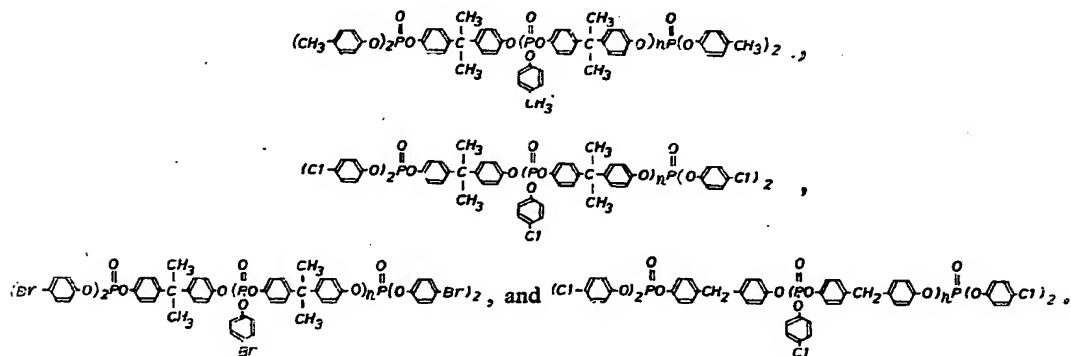


(wherein X, m and n are as previously defined).

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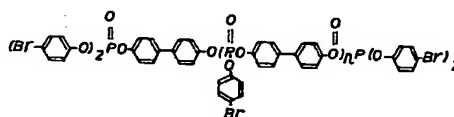
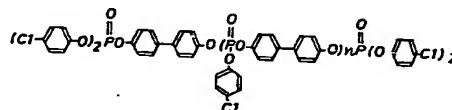
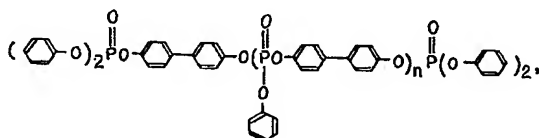
12. A polyester composition according to Claim 11, wherein the phosphorus compound is selected from the following group:

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$$X_m \text{---} (\text{C}_6\text{H}_4\text{---O})_2 \text{P}(=\text{O})(\text{C}_6\text{H}_4)_2 \text{O} \left[ \text{P}(=\text{O})(\text{C}_6\text{H}_4)_2 \text{O} \right]_x \text{P}(=\text{O})(\text{C}_6\text{H}_4)_2 \text{O} \text{---} X_n$$

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